



Me, Wave-email-notifications@appspot.com and Embeddy:

Dec 3, 2009 ▼

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Ugi Product 233 and 234 Characterization by FT-NMR and FT-IR



Go

[UsefulChemistry 233](#)

[UsefulChemistry 234](#)

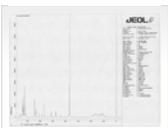
Results

H NMR of Ugi 233 (1.4) with TMS: [233hnmrtms](#)

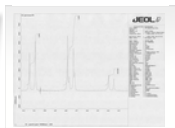
H NMR of Ugi 234 (2.1) with TMS: conversion to .jdx unsuccessful

H NMR of Ugi 233 (1.4) with TMS converted by Antony Williams: [233hnmrtmsjdx](#)

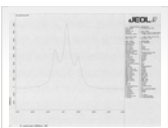
H NMR of Ugi 234 (2.1) with TMS converted by Antony Williams: [234hnmrtmsjdx](#)



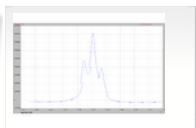
[Ugi1.4tms.pdf](#)



[Ugi1.4tmsdoublet.pdf](#)

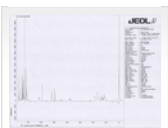


[Ugi1.4tmsclose.pdf](#)

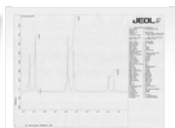


[Ugi1.4jspecttmsclose.pdf](#)

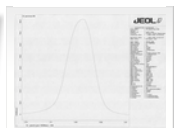
Note: These two close-ups seem to show the compression distorting the resolution. The peaks look the same, except they are closer together and the bottoms are distorted in the JspecViewer. And if one looks close enough, one can even see where the spectrum was shifted together causing the compression and distortion.



[Ugi2.1tms.pdf](#)



[Ugi2.1tmsdoublet.pdf](#)



[Ugi2.1tmsclose.pdf](#)

Original Delta 1D data files:



[dab_ugi_1_4_h1tms_12_01_09.1](#)



[dab_ugi_2_1_h1tms_12_01_09.1](#)

Log

12.01.09

15.30 Washed NMR tubes with CHCl₃ and vacuum dessicated

16.00 Added 7 uL TMS to about 1 mL of Ugi 2.1 in CDCl₃ solution

16.17 Auto-gradient shim and lock (Z1:-1196,Z2:-1016,Z3:-152,Z4:800)

16.21 Proton NMR (128 scans) of Ugi 2.1 (234) (Autogain: 17)

16.48 Added 4 uL TMS to about 0.7 mL of Ugi 1.4 (233) in CDCl₃

16.50 Auto-gradient shim and lock (Z1:-1303,Z2:-1063,Z3:-223,Z4:781)

16.54 Proton NMR (128 scans) of Ugi 1.4 (233) (Autogain: 14)

Me: I did not notice that the carbonyl by the triple bond resonated to form the double bond to the nitrogen. I now see that this is what Khalid meant by the [E and Z rotamers](#). I plan on performing another HyperNMR prediction with this accounted for, and analyze the spectra for these E and Z rotamers. Nov 30, 2009 ▼

Jean-Claude: I am not sure that the prediction software can even handle diastereotopic protons - ACDLabs failed to do that so be careful with prediction software. Nov 30, 2009 ▼

Jean-Claude: I made a suggestion on the wiki for easily getting your rotamer ratio Nov 30, 2009 ▼

Me: Thank you, I should be able to find the rotamer ratio easily. The prediction software can only give me shielding, tau, and chemical shifts with these molecules, since they are larger than 20 NMR atoms. However, it calculates it for all the atoms in the molecule. I will heed your warning and not trust the results of the prediction much yet. Dec 1, 2009 ▼

Khalid: David- Your HNMRs for both UC233 and UC234 are really clean and nice. However, like Dr. Bradley mentioned earlier they seem to be a little shifted. You can compare UC233 with a very similar compound that I obtained from UCExp173G, this compound is similar except for cyclohexylgroup. Nov 30, 2009 ▼

I suggest it might be best if you rerun the HNMRs for both UC233 and UC234 in CDCl₃ with TMS in it. If you do not have CDCl₃ spiked with TMS, I can send a 100mL bottle.

Again good job on isolating the two novel Ugi products.

Me: Khalid, thank you for recommending that I try cyclohexylisocyanide and for teaching me how to perform the Ugi 4-component reaction. I can spike the solutions with TMS myself. I might be able to remove the TMS and chloroform to recover the solid Ugi Product by vacuum dessicating it. Dec 1, 2009 ▼

Jean-Claude and me: David the TMS is a standard way to zero the NMR because it evaporates easily - just add very little. Your NMR look really good - once you have the right scale we can wrap up very quickly. Dec 1, 2009 ▼

Me: Great! I have scheduled NMR time tomorrow afternoon when I will add maybe less than a drop of TMS to the solutions of Ugi product in CDCl₃.

Dec 1, 2009 ▼

Try adding 5-10 microliters. 💬

Me: Okay ... so 5-10 microliters would still be less than a drop.

Dec 1, 2009 ▼

Absolutely - your NMRs have clear signals when you put all the material in. As a reference one drop is about 50 microliters. If you have too much TMS it might warp your sample. 💬

Me: I've been using a micropipet anyway for the TMS in other samples for Cl.

Dec 1, 2009 ▼

Cool -so you know what to expect. 💬

Me: Yeah.

Dec 1, 2009 ▼

Perfect - I'll wait for you to upload the next NMR and we can discuss in detail. 💬

Me: Sounds good. I don't have the original reactants. But I should be able to confirm the purity somewhat.

Dec 1, 2009 ▼

Your Ugi products look pretty pure to me - were you able to get the rotamer ratio? 💬

Me: I have not looked into that yet.

Dec 1, 2009 ▼

It is easy - there are 2 sets of doublets around 4 ppm - these are the diastereotopic CH₂ from the benzyl. Just take the ratio of the big doublet to small doublet. 💬

Me: I am doing that right now. It is exactly 5:1.

Dec 1, 2009 ▼

That sounds about right. 💬

Me: I wonder if it will be the same in the runs tomorrow.

Dec 1, 2009 ▼

It should be if you have the same solvent and same concentration. 💬

Me: The solvent concentration might have decreased due to evaporation, but the relative concentration should be the same.

Dec 1, 2009 ▼

Yes the concentration won't have much impact on the ratio - I would think most of the CDCl₃ has evaporated by now - 💬

Me: I can always add more CDCl₃. And besides, a higher concentration might give a better NMR.

Dec 1, 2009 ▼

yes definitely add more so you have at least 0.7 ml in the tube for good results. 💬

Me: I've been scaling it to the area the NMR scans to make sure all of the signal passes through solution.

Dec 1, 2009 ▼

Just be careful you have enough liquid - if you don't you'll get bad spectra. 💬

Me: The Ugi Products actually dissolve surprisingly well in chloroform.

Dec 1, 2009 ▼

You can check the solubility of Ugi products in general on our ONSC database - yet chloroform is good for amides. 💬

Me: So it is expected that they dissolve well in chloroform?

Dec 1, 2009 ▼

Yes unless you have phenanthrene. 💬

Me: okay.

Dec 1, 2009 ▼

OK good luck - email me if you have any problems 💬

Me: I will do that.

Dec 1, 2009 ▼

Me: The H NMR with TMS on Ugi 2.1 had large peaks for chloroform and TMS (very large peak) to aid in chemical shift determination. And the H NMR with TMS on Ugi 1.4 had a large peak for TMS that was about the same size at the methoxy peaks. The interactive spectra is not as detailed as the original spectra using the JEOL software. I think the conversion from .nmr to .jdx causes some loss in resolution. The compression of the spectra is also probably due to the conversion process.

Dec 2, 2009 ▼

Jean-Claude: what software are you using for the conversion?

Dec 3, 2009 ▼

Me: Delta 1D export by JEOL











Dec 3, 2009 ▼

Tony27587@googlewave.com: David..if you can send me the original JEOL file let me process on my end and see what I get. It may be buried in the Wiki or on here but did you compare the Jeol printout and the JDX..are they different? I recall seeing a printout somewhere and that they were different. Just checking.

Dec 3, 2009 ▼

Me: I have attached the original Delta 1D data files. And yes, the printout and JDX files are different in that the JDX files are compressed and of lower resolution.

Dec 3, 2009 ▼

	Me: Dr. Williams, thank you for converting the spectra to JDX from the JEOL file. The spectra are no longer compressed and have the same resolution as the JEOL print-outs. This confirms that the loss in resolution and compression of spectra was a result the automated conversion by JEOL.	Dec 3, 2009 ▼
	Jean-Claude: David - do you have the procedure for converting the files correctly from now on?	Dec 4, 2009 ▼
	Me: Dr. Bradley, I do not have the procedure. Dr. Williams, how did you convert the files and is there a way for me to convert them in the future?	Dec 4, 2009 ▼
	Jean-Claude: khalid are you able to convert the files properly?	Dec 7, 2009 ▼
	Tony27587@googlewave.com: I loaded the original files into ACD/NMR processor and then simply processed them and exported as JCAMP files.	Dec 8, 2009 ▼
	Me: I've just downloaded ACD/NMR processor 12.0. I understand how to import and process. However, there are several export options. How exactly did you export the spectra as JCAMP files?	Nov 15, 2010 ▼
	Me: It looks like you exported as JCAMP-DX real PAC form. This seems to work for me. This is a great way to convert the spectra! Thanks again!	Nov 15, 2010 ▼
	Me: Actually, it seems like any of the JCAMP-DX export settings work! By the way, the file can be integrated in SAMS by opening the file in JSpecView and saving as JCAMP-DX.XY.	Nov 15, 2010 ▼
	Tony27587@googlewave.com: ANy of the JCAMP formats will work for sure... I use SQZ commonly.	Nov 15, 2010 ▼
	Andrew: Good work david	Nov 15, 2010 ▼